



**NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

GB 23200.67-2016

Replacing SN/T 2647-2010

**Food safety national standard -- Determination of residual
alkynyl chloride residues in foodstuffs by gas
chromatography-mass spectrometry**

食品中炔苯酰草胺残留量的测定 气相色谱-质谱法

(English Translation)

Issued on: December 18, 2016

Implemented on: June 18, 2017

**Issued by: National Health and Family Planning Commission of the
People 's Republic of China**

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Foreword

This standard replaces SN/T 2647-2010 "Determination of the amount of acetylene acenamide residues in import and export food by gas chromatography-mass spectrometry".

Compared with SN/T 2647-2010, the main changes are as follows.

- Standard text format is modified to national standard text format for food safety;
- the name and scope of the "import and export food" to "food";
- increase the "other food reference implementation" in the standard range.

This standard replaced the previous version of the standard release.

-SN/T 2647-2010.

National standards for food safety Determination of residual alkynyl chloride residues in foodstuffs by gas chromatography - mass spectrometry

1 Scope

This standard specifies the method of gas chromatography-mass spectrometry for the separation of acetophenone residues in food.

This standard applies to spinach, carrots, strawberries, peanuts, chicken, onions, cod, honey, chestnut and tea the detection and confirmation of residues, other food can refer to the implementation.

2 Normative reference documents

The following documents are indispensable for the application of this document. For dated references, only the date of the note applies this document. For undated references, the latest edition (including all modifications) applies to this document.

GB 2763 National Standard for Food Safety - Maximum Residue Limit of Pesticides in Foodstuffs GB/T 6682 Analytical laboratory water specifications and test methods

3 Principle

The sample was extracted with acetonitrile and purified by liquid-liquid partitioning and propyl ethylenediamine-bonded silica gel (PSA) solid phase extraction column.

Determination of mass spectrometry by gas chromatography - electron bombardment and external standard method.

4 Reagents and materials

Unless otherwise specified, the reagents used are of analytical grade and the water is in accordance with the primary water specified in GB/T 6682.

4.1 Reagents

4.1.1

Acetone (C₃H₆O). Pesticide Residue.

4.1.2

acetonitrile (C₂H₃N). pesticide residues.

4.1.3

N-Hexane (C₆H₁₄). Pesticide Residue.

4.1.4

sodium chloride (NaCl). 650 °C burning 4 h, placed in a dryer cooling, standby.

4.2 Solution preparation

4.2.1

acetonitrile saturated n-hexane. take 100mL n-hexane, 100mL acetonitrile in the separatory funnel, the oscillation mix, take the upper stand.

4.2.2

n-hexane saturated acetonitrile. take 100mL n-hexane, 100mL acetonitrile in the separatory funnel, shake mix, take the next stand.

4.2.3

Acetone-n-Hexane (3 7, V/V). Take 300 mL of acetone and add 700 mL of n-hexane to shake.

4.3 Standards

4.3.1

Acetylacetamide standard substance (propylamide, molecular formula. $C_{12}H_{11}Cl_2NO$, molecular weight 255, CAS 5.2 solid phase extraction device.

5.3 homogenizer, speed 10000 r/min.

5.4 Rotary Evaporator.

5.5 nitrogen concentrator.

5.6 with a plug centrifuge tube. 50 mL, 100 mL.

5.7 Concentrated bottles. 50 mL, 100 mL.

5.8 Analytical balance. 0.01 g and 0.0001 g.

6.1.1 Grains and nuts

Take a representative sample volume of 500g, all ground and pass through a 2.0 mm round hole screen. Mix well, into a clean container, sealed, Mark the mark.

6.1.2 Fruits and vegetables

Take a representative sample 500g, chopped (not washable), with a food mashed machine to sample into a slurry. Mix well and put it clean of the container, sealed, marked mark.

6.1.3 Meat and fish

Take a representative sample 500g, fully crushed evenly, into a clean container as a sample. Sealed, marked.

6.1.4 Honey

Take a representative sample 500g, the crystallization of honey samples will be evenly stirred; on the crystallization of honey samples, in the closed the case, the sample bottle placed in not more than 60 °C in the water bath in the warm, shaking, until the sample all melted and stir well, quickly cooled to room temperature, in the melting must pay attention to prevent moisture evaporation. Load a clean container, sealed, marked with a mark.

6.1.5 Tea

Take a representative sample of 500 g, grind all with an attritor and pass through a 2.0 mm round hole screen. Mix well and clean the clean container Inside, sealed, marked mark.

Note: The above sample sampling site according to GB 2763 Appendix A implementation.

6.2 Sample storage

Grains and nuts are stored at 0 to 4 ° C; other samples are cryopreserved at -18 ° C or lower.

During the operation of the sample preparation, the sample should be protected from contamination or changes in the content of the residue.

7.1 Extraction

Weigh 10 g of sample (accurate to 0.01 g) in a 100 mL stoppered centrifuge tube, add 10 mL of water, add 40.0 mL Hexane saturated acetonitrile, homogenizer extraction 2 min (honey plus water 10mL and acetonitrile to be vigorous oscillation 20min), then add 5g sodium chloride, Severe oscillation 10min, 3000 r/min centrifugal 10min, to be purified.

7.2.1 Liquid/liquid distribution purification

Take the upper extract 20.0 mL (spinach, carrot, strawberry) or 10.0 mL (peanuts, chicken, onions, cod, honey, chestnut and tea) were transferred to a 50 mL stoppered centrifuge tube, 10 mL of acetonitrile was added to the saturated n-hexane, shaken for 3 min, left to stand, discarded the n-hexane phase was removed and the n-hexane phase was discarded again with 10 mL of acetonitrile saturated n-hexane. The lower acetonitrile phase was collected in 100 mL Concentrated in a 40 ° C water bath and concentrated to approximately 1 mL.